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भारतीय मानक

ऐसीफेट, तकनीकी — विशिष्टि

Indian Standard

ACEPHATE, TECHNICAL — SPECIFICATION

(First Reprint NOVEMBER 1994)

UDC 632.952 CAR

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

August 1990 **Price Group 1**

AMENDMENT NO. 1 DECEMBER 1992
TO
IS 12915 : 1990 ACEPHATE, TECHNICAL — SPECIFICATION

(*Page* 2, *clause* A-1.1) — Substitute the following for the existing clause:

'A-1.1 Two methods have been specified for the determination of acephate content, namely, gas chromatographic (GLC) method (*see* A-2) and phosphorus method (*see* A-3). The GLC method shall be followed by independent laboratories. The phosphorus method shall be followed by the manufacturer only for routine in-plant quality control checks.'

(*Page* 2) — Insert the following at the end:

'A-3 PHOSPHORUS METHOD

A-3.1 Apparatus

A-3.1.1 *Shaking machine*

A-3.2 Reagents

A-3.2.1 *Ammonium Nitrate Solution* — Dissolve 100 g ammonium nitrate (free of phosphates) in water and dilute to 1 000 ml.

A-3.2.2 *Methyl Orange Indicator Solution* — 0.1 percent aqueous solution (*v*/*v*).

A-3.2.3 *Standard Sodium Hydroxide Solution* — 0.1 N.

A-3.2.4 *Standard Hydrochloric Acid Solution* — 0.1 N.

A-3.2.5 *Phenolphthalein Indicator* — 1 percent solution in ethyl alcohol.

A-3.2.6 *Ammonium Hydroxide Solution* — Aqueous solution (1:3; *v*/*v*).

A-3.2.7 *Nitric Acid Solution* — Aqueous solution (1:3;*v*/*v*).

A-3.2.8 *Ammonium Molybdate Solution* — Dissolve 100 g ammonium molybdate in a mixture of 144 ml ammonium hydroxide and 271 ml water. Cool to room temperature and add slowly with constant stirring to a cool mixture of 489 ml nitric acid and 1148 ml water. Allow the mixture to stand for 48 hours in a stoppered vessel. Filter immediately before use and add 5 ml nitric acid to each 100 ml of filtrate.

A-3.2.9 ***Chloroform***

A-3.2.10 ***Perchloric Acid*** — 70-72 percent (*v/v*).

A-3.3 Procedure

A-3.3.1 Accurately weigh the sample containing 200 mg phosphorus and extract with 200 ml chloroform by shaking for one hour on a shaking machine. Filter into a 250-ml volumetric flask, wash the residue twice with 20 ml portions of chloroform and make up the volume with chloroform.

A-3.3.2 Transfer the aliquot into a 250-ml beaker, add 30 ml nitric acid, dropwise at first until vigorous reaction is completed, boil gently for 30 minutes. Cool and add perchloric acid.

A-3.3.3 Boil gently the solution at A-3.3.2 until it is colourless or nearly so and appearance of dense fumes. Do not boil the solution to dryness. The fuming temperature shall be at about 170°C for at least 30 minutes. Add more quantity of perchloric acid, if necesary. Cool slightly, add 50 ml water and boil for a few minutes.

A-3.3.4 Add to the solution (A-3.3.3), 20 ml ammonium nitrate solution and 1.0 ml methyl orange indicator. Adjust the *p*H to just alkaline by adding ammonium hydroxide and then to acidic with addition of nitric acid. Dilute to 100 ± 20 ml with distilled water.

A-3.3.5 Add ammonium molybdate solution (A-3.2.8) to the solution (A-3.3.4) and agitate continuously for 15 minutes. Allow the precipitate to settle for one or two minutes.

A-3.3.6 Decant the supernatant liquid as at A-3.3.5 through a glass funnel fitted with a pad of macerated asbestos approximately 0.31 cm thick on a perforated disc and filter under vacuum. Transfer the precipitate to the funnel and rinse the beaker several times with a stream of water. Wash the precipitate 5 times with 25-30 ml water. Transfer the precipitate, pad and perforated disc back into the 250-ml beaker and rinse the funnel with not more than 100 ml water.

A-3.3.7 Add sufficient standard sodium hydroxide to completely discharge the yellow phospho-molybdate and then add 2-3 ml in excess. Add 0.4 ml phenolphthalein indicator. Rinse the sides of the beaker with 5-10 ml sodium hydroxide. Titrate with standard hydrochloric acid to a distinct light pink colour and point and verify the end point at 7.85 ± 0.05 *p*H by means of a *p*H meter.

A-3.4 Calculation

$$\text{Phosphorus content, percent by mass } P = \frac{(V_1 \times N_1) - (V_2 \times N_2)}{M} \times 0.134\,9$$

where

V_1 = volume, in ml, of standard sodium hydroxide;

N_1 = normality of standard sodium hydroxide;

V_2 = volume, in ml, of standard hydrochloric acid

N_2 = normality of standard hydrochloric acid; and

M = mass, in g, of sample in aliquot (A-3.2.2).

Acephate content, percent by mass = $P \times 5.91$'.

(FAD 1)

AMENDMENT NO. 2 DECEMBER 1995
TO
IS 12915 : 1990 ACEPHATE, TECHNICAL — SPECIFICATION

(*Page* 1, *clause* 2.1) — Substitute '1070 : 1992 Reagent grade water (*third revision*)' *for* '1070 : 1977 Water for general laboratory use (*second revision*)'.

(*Page* 1, *clause* 7.2) — Substitute '1070 : 1992' *for* '1070 : 1977'.

(*Amendment No.* 1, *clause* A-3.3.2) — Substitute the following for the existing:

'Transfer 50 ml aliquot into a 250-ml beaker, add 30 ml nitric acid dropwise at first until vigorous reaction is completed, boil gently for 30 minutes. Cool and add perchloric acid.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 3 MAY 1998
TO
IS 12915 : 1990 ACEPHATE, TECHNICAL — SPECIFICATION

[*Page* 2, *clause* A-1.1, *second sentence* (*see also Amendment No.* 1)] — Insert the following at the end:

'or for plant quality control checks as referee method'.

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 4 APRIL 2002
TO
IS 12915 : 1990 ACEPHATE, TECHNICAL — SPECIFICATION

(*Page* 2, *clause* **A-2.1.1**, *line* 7) — Substitute '100 cm length' *for* '38 cm length'.

(*Page* 2, *clause* **A-2.3.1**, *line* 1) — Substitute 'di-butyl phthalate' *for* 'di- isobutyl phthalate'.

(FAD 1)

Reprography Unit, BIS, New Delhi, India

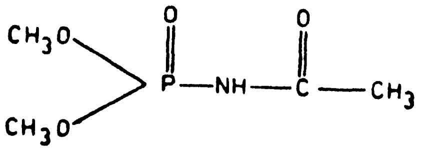
Pesticides Sectional Committee, FADC 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 25 January 1990, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agricultural Division Council.

Acephate, technical is employed in the preparation of insecticidal formulations.

Acephate is the accepted common name by ISO for O, S-dimethyl acetyl phosphoramidothiote. The empirical and structural formula and molecular mass are as follows:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_4H_{10}NO_3PS$		183·16

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ACEPHATE, TECHNICAL — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for acephate, technical.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1977	Specification for water for general laboratory use (*second revision*)
6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
8190 (Part 1) : 1988	Requirements for packing of pesticides: Part 1 Solid pesticides (*second revision*)
10946 : 1984	Methods of sampling for technical-grade pesticides

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white free-flowing powder, free from extraneous impurities or hard lumps.

3.2 The material shall also comply with the requirements specified in Table 1.

4 PACKING

4.1 The material shall be packed in fibre or mild steel drums provided with low density polyethylene liner. The general requirements as given in IS 8190 (Part 1) : 1988 shall also be followed.

5 MARKING

5.1 The container shall bear legibly and indelibly the following information and any other information as is necessary under the *Insecticides Act*, 1968 and Rules:

a) Name of the material;
b) Indication of the source of manufacture;
c) Batch number;
d) Date of manufacture and expiry;
e) Acephate content, percent (*m/m*);
f) Net mass of the contents; and
g) The cautionary notice as worded in the *Insecticides Act*, 1968 and Rules.

Table 1 Requirements for Acephate Technical

(*Clauses* 3.2 *and* 7.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Clause No. of IS 6940 : 1982
(1)	(2)	(3)	(4)	(5)
i)	Acephate content, percent by mass, *Min*	97·0	A	—
ii)	Melting point, °C	86-88	—	6
iii)	Material insoluble in water, percent by mass, *Max* (*see* Note)	1·5	—	9
iv)	Acidity (as H_2SO_4), percent by mass, *Max*	1·5	—	11·3

NOTE — Water shall be used instead of acetone as the solvent.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 10946 : 1984.

7 TESTS

7.1 Tests shall be carried out as referred to in col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Item* (i)]

DETERMINATION OF ACEPHATE CONTENT

A-1 GENERAL

A-1.1 For the determination of acephate content a gas chromatographic (GLC) method has been specified.

A-2 GLC METHOD

A-2.1 Apparatus

A-2.1.1 *Gas Liquid Chromatograph (GLC)*

A GLC unit fitted with a hydrogen flame ionization detector and also a printer-plotter-*cum*-integrator is used for this determination. The suggested operative parameters are as follows, but can be changed in any other equipment, provided standardization is done:

Column	38 cm length and 2 mm ID Glass packed with 3 percent OV-225 on Gas Chrom Q (120-140) mesh, conditioned at 250°C overnight	
Temperature	Column oven	190°C
	Injector	250°C
	Detector	250°C
Carrier Gas	Nitrogen	
Flow Rate	30 ml/minute	

NOTE — The attenuation is to be adjusted for obtaining peaks at 60-80 percent of full-scale.

A-2.1.2 *Microlitre Syringe*

A-2.2 Reagents

A-2.2.1 *Internal Standard Solution*, Di-isobutyl phthalate. AR grade or GLC grade.

A-2.2.2 *Methylene Chloride*, AR grade.

A-2.2.3 *Standard Acephate*, of known purity.

A-2.3 Procedure

A-2.3.1 *Preparation of Internal Standard Solution*

Weigh 0·2989 ± 0·010 g of di-isobutyl phthalate into a 1-litre volumetric flask and make up the volume with methylene chloride.

A-2.3.2 *Preparation of Standard Solution*

Weigh accurately 0·100 0 g of acephate standard into a 100-ml volumetric flask and make up the volume with internal standard solution (A-2.3.1).

A-2.3.3 *Preparation of Sample Solution*

Weigh accurately a quantity of sample equivalent to 0·100 0 g of acephate into a 100-ml volumetric flask and make up the volume with internal standard solution (A-2.3.1). Mix well to dissolve the acephate and centrifuge down the remaining solids.

A-2.3.4 *Calibration*

Inject simultaneously 1·0 µl of internal standard (A-2.3.1) and standard (A-2.3.2) solutions into the GLC and from the integrator print out and note down the peak areas of acephate and internal standard. Adjust the attenuation in such a way that acephate and internal standard peaks are obtained at 60-80 percent of full-scale in all the cases. Calculate the ratio of peak areas of acephate standard and internal standard.

A-2.3.5 *Estimation*

Inject 1·0 µl of sample solution (A-2.3.3) into the GLC and calculate the ratio of peak areas of sample and internal standard.

A-2.4 Calculation

$$\text{Acephate content, percent} = \frac{R_s \times W_{st} \times P}{R_{st} \times W_s}$$

where

R_s = average ratio of peak areas of acephate sample and internal standard;

R_{st} = average ratio of peak areas of acephate standard and internal standard;

W_{st} = mass, in g, of acephate standard;

W_s = mass, in g, of acephate sample; and

P = percent purity of standard acephate.

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Amendments Issued Since Publication

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